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(54)【発明の名称】　無機箔粉の製造方法および無機箔粉

(57)【要約】
【課題】　厚さが極めて薄く、しかも金属光沢等に優れた金属箔粉等の無機箔粉とその製造方法を提供する。
【解決手段】滑剤等の添加物の層をポリオレフィンフイルムの面に広く形成せしめ、該添加物の層上に無機蒸着薄膜層を形成・積層し、後該添加物の滑性や可溶解性や剥離性を利用して、溶媒等による溶媒処理や機械的剥離を施し容易にしかも収率よく該無機蒸着薄膜層を剥離して粉砕する方法とそれによって得られる厚さが極めて薄く、しかも金属光沢等に優れた金属箔粉等の無機箔粉。

【特許請求の範囲】
【請求項１】ポリオレフィンフイルム（Ａ）の面上に、厚さが０．００５μｍ～３μｍの無機蒸着薄膜層（Ｃ）を設け、該無機蒸着薄膜層（Ｃ）を剥離し、剥離された層（Ｃ）を破砕する無機箔粉の製造方法において、該ポリオレフィンフイルム（Ａ）に予め含有保持せしめられた添加物（Ｂ）の層を、およびまたは該同一添加物（Ｂ）の層を、剥離層としてポリオレフィンフイルム（Ａ）の面と無機蒸着薄膜層（Ｃ）との間に形成せしめることを特徴とする無機箔粉の製造方法。
【請求項２】無機蒸着薄膜層（Ｃ）がアルミニウム、銀、金、ニッケル、クロム、錫、銅、亜鉛、インジウム、チタン、マグネシウムの単体金属又はこれらの合金およびそれらの混合物の少なくとも一種からなる請求項１記載の無機箔粉の製造方法。
【請求項３】無機蒸着薄膜層（Ｃ）がアルミニウム、錫、インジウム、チタン、ケイ素、亜鉛、マグネシウムの酸化物、炭化物、窒化物、硫化物、ハロゲン化物の単体化合物または複合化合物の少なくとも一種からなる請求項１記載の無機箔粉の製造方法。
【請求項４】請求項１から３に記載の製造方法によって製造された、厚さが０．００５μｍ～３μｍ、大きさが平均２μｍ～１５０μｍであることを特徴とする無機箔粉。
【発明の詳細な説明】
【０００１】
【発明の属する技術分野】本発明は、少なくとも無機蒸着薄膜層の破砕非定型偏平片または破砕半定形（例えば矩形）偏平片である無機箔粉の製造方法に関するものであり、該無機箔粉をインク、塗料等の被覆材および充填剤として使用することによって、対象物に表出された印字や描画にまたは表面全体にまたは被充填材に、金属箔粉の場合には金属光沢等を、金属化合物箔粉の場合は該箔粉の保有する機能を付与し得る無機箔粉の製法および無機箔粉に関する。
【０００２】
【従来の技術】金属箔粉として、①古くから知られている貴金属等の微粉、即ち展延により得られた展延箔の破砕片、②近年の蒸着時の凝集微細片、また③金属箔の破砕片である金属箔粉、④特公平５－７７６４３号公報に記載のフイルム上の蒸着金属からの裁断グリッター、また⑤特公昭５３－３５１３５号公報に記載の片面または両面が樹脂で被覆された金属蒸着箔の破砕小片である金属粉等が知られている。
【０００３】
【発明が解決しようとする課題】前記従来の金属箔または金属粉は、①古くから知られている貴金属等の微粉、即ち展延により得られた展延箔の破砕片は、その貴金属の耐腐食性、美麗さ、微細であることから多用されてはいるが、その製法から高コストであり、大量に生産できない、比重が大きすぎて分散媒体中で沈殿しやすいなどの課題を有している。
②近年の蒸着時の凝集微細片は、その微細さにおいて魅力のあるものだが、高コスト、微細過ぎて、金属光輝性の美麗さを得にくい、取り扱いにも難点を有しているなどの課題を有している。
【０００４】③金属箔の破砕片である金属箔粉は、比較的低コストで入手でき、光輝性においてもある程度の性能を有しており多用されているが、その大きさが数十μｍ～数百μｍのものがほとんどであり、塗布面の平滑さや高光輝性を、より要求される場合には対応し得ない等の課題を有している。
④特公平５－７７６４３号公報に記載のフイルム上の蒸着金属からの裁断グリッターは、特定の用途においてはその低コストから使用されるが、大きさは数ｍｍ程度の大きさであり、用途が限定される。
⑤特公昭５３－３５１３５号公報に記載の片面または両面が樹脂で被覆された金属蒸着箔の破砕小片である金属粉は、その大きさ、比較的低コストから、その高い光輝性により多用されているが、樹脂で被覆されているため、樹脂層厚さのためにより厚くなり厚さの点においてや、耐熱性において限界がある。さらにフイルム上に可溶解性ポリマーの層を形成し、金属蒸着薄膜層を形成し可溶解性ポリマーの層を溶解処理することで、金属蒸着薄膜層をフイルムから剥離し、破砕して金属箔粉を得る方法も知られているが、金属箔粉から該箔粉に付着した可溶解性ポリマーの層を除去することに多大のコストおよびまたは高額な危険物取り扱い用の設備を要する等の課題を抱えていた。本発明は、前記従来の金属箔粉製法の抱える課題を解決せんとするものであり、厚さが３μｍ以下で、大きさも数μｍ～百μｍ程度の、光輝性に優れ、耐熱性に優れた箔粉を提供せんとするものである。
【０００５】
【課題を解決するための手段】すなわち本発明は、ポリオレフィンフイルム（Ａ）の面上に、厚さが０．００５μｍ～３μｍの無機蒸着薄膜層（Ｃ）を設け、該無機蒸着薄膜層（Ｃ）を剥離し、剥離された層（Ｃ）を破砕する無機箔粉の製造方法において、該ポリオレフィンフイルム（Ａ）に予め含有保持せしめられた添加物（Ｂ）の層を、およびまたは該同一添加物（Ｂ）の層を、剥離層としてポリオレフィンフイルム（Ａ）の面と無機蒸着薄膜層（Ｃ）との間に形成せしめることを特徴とする無機箔粉の製造方法であり、また無機蒸着薄膜層（Ｃ）がアルミニウム、銀、金、ニッケル、クロム、錫、銅、亜鉛、インジウム、チタン、マグネシウムの単体金属又はこれらの合金およびそれらの混合物の少なくとも一種からなる前記の無機箔粉の製造方法であり、また無機蒸着薄膜層（Ｃ）がアルミニウム、錫、インジウム、チタン、ケイ素、亜鉛、マグネシウムの酸化物、炭化物、窒化物、硫化物、ハロゲン化物の単体化合物または複合化

合物の少なくとも一種からなる前記の無機箔粉の製造方法であり、さらにまた前記何れかの製造方法によって製造された、厚さが０．００５μｍ～３μｍ、大きさが平均２μｍ～１５０μｍであることを特徴とする無機箔粉である。

【０００６】

【発明の実施形態】本発明における、ポリオレフィンフイルム（Ａ）としては、蒸着工程の加熱、真空、応力等に耐え得る自立性のポリオレフィンフイルムであれば特に限定されないが、ポリエチレン、ポリプロピレン等のホモポリオレフィンフイルムやこれらのポリオレフィン系共重合体からのフイルム等が挙げられるが、なかでもポリプロピレン系フイルムとポリエチレン系フイルムが好ましく使用できる。これらのフイルムの厚さは４μｍから１００μｍであり、４μｍに満たないものは、工程等で取り扱い性に問題が多く、１００μｍを超えると、柔軟性に乏しくなり、剥離等に問題が多くなる。更にこれらのポリオレフィンフイルム（Ａ）としては、単層のフイルムでもよく、例えば共押し出し法による複層フイルムであってもよい。またこれらのフイルムは、無延伸、一軸延伸、二軸延伸、弱一軸延伸等の製法による限定を特に受けるものではない。

【０００７】本発明におけるポリオレフィンフイルム（Ａ）としては、該フイルムに滑剤やステイッキング防止剤、防曇剤、帯電防止剤等としての添加物（Ｂ）が、添加・含有・散布等の手段で含有、保持せしめられ、該添加物を０．０１～２．０重量％の範囲で該フイルムが有するものである。添加物（Ｂ）の層としては、ポリオレフィンフイルム（Ａ）上にブリードアウト、塗布等で形成され、その層上に形成される厚さが０．００５μｍ～３μｍの無機蒸着薄膜層（Ｃ）の形成時に蒸散したり、熱変性したりして金属箔粉の製造に支障をきたさないもので、分子量が５０００以下のもの、好ましくは３０００以下さらに好ましくは１０００程度以下の化合物が好ましく、無機蒸着薄膜層（Ｃ）のポリオレフィンフイルム（Ａ）からの溶解等による剥離のし易いものであればよく、特に限定されないが、脂肪酸、金属脂肪酸、低分子界面活性剤、ポリオキシアルキレングリコール、パラフィン、酸アミド、脂肪酸エステル等およびこれらの変性物を主成分とするものが挙げられ、またこれらと無機化合物との変性物、および混合物等も使用できる。本発明における「該ポリオレフィンフイルム（Ａ）に予め含有保持せしめられた添加物（Ｂ）の層を、およびまたは該同一添加物（Ｂ）の層を、剥離層としてポリオレフィンフイルム（Ａ）の面と無機蒸着薄膜層（Ｃ）との間に形成せしめること」は、特に限定されないが、塗布、散布等でフイルム上に蒸着前に形成してもよく、添加物（Ｂ）を含有せしめた同種ポリマーを共押し出しなどで該添加物（Ｂ）を殆ど含まない同種ポリマー上に積層（複層）したフイルムとし所謂ブリードアウトせしめたものでもよく、また予めポリオレフィンフイルム（Ａ）である基材フイルムに練り込んでおき蒸着前または蒸着後に、経時処理や、熱経時処理や熱処理等によって該フイルム表面にブリードアウトせしめる方法等が挙げられる。予めポリオレフィンフイルム（Ａ）である基材フイルムに練り込んでおき蒸着前または蒸着後に、経時処理、熱経時処理や熱処理等によって該フイルム表面に所謂ブリードアウトせしめる方法においては、無機蒸着薄膜層を蒸着した後に所謂ブリードアウトせしめる方法がより好ましく採用され、その中でも４０℃以上の加熱を伴う熱経時処理が好ましく、該フイルム表面に該添加物層が均一に形成されることになる。また蒸着前に経時処理、熱経時処理や熱処理を施して該フイルム表面に含有添加物を所謂ブリードアウトせしめる方法においては、該フイルム表面に該添加物層が均一に形成されることが好ましく、好ましくは無機蒸着薄膜層（Ｃ）の剥離が５０％以上の面積で容易になるように、５０％以上に形成されること、より好ましくは７０％以上、さらに好ましくは８５％以上であり、５０％以下の場合には容易に剥離するものが５０％以下の面積分となり箔粉製造における収率の低下、コストの上昇を招くことになる。したがって、本発明は製造後の放置による経時で微小な部分的添加物（Ｂ）の層を形成されたものでなく、前記した広範囲に添加物（Ｂ）の層が形成されたポリオレフィンフイルム（Ａ）を選択使用することを含むものである。

【０００８】本発明における厚さが０．００５μｍ～３μｍの無機蒸着薄膜層（Ｃ）の形成方法は蒸着によるものであれば特に限定されず加熱蒸着、スパッタリング、等から適宜選択使用すればよい。これらの無機蒸着薄膜層（Ｃ）は、金属蒸着薄膜層の単層でもよく、異種または同一種の複層でもよく、無機化合物蒸着薄膜の単層でもよく、異種または同一種の複層でもよい。また、金属蒸着薄膜層と無機化合物蒸着薄膜層との積層でもよい。無機蒸着薄膜層（Ｃ）のうち金属蒸着薄膜層として使用される金属は、金属光沢を有する等の機能を有するものであれば特に限定されるものではないが、アルミニウム、銀、金、ニッケル、クロム、錫、亜鉛、インジウム、チタン、の単体金属又はこれらの合金およびそれらの混合物の少なくとも一種以上が適宜選択使用される。また、前記例示のものの中から一種または二種以上を適宜選定し一層または二層以上にして使用してもよい。これらの金属蒸着薄膜層の厚さは、０．００５μｍ～３μｍの範囲が好ましく、０．００５μｍに満たないときは、光輝性においてまた機能性においてその性能は乏しく、３μｍを超えるときは、これ以上厚さを大きくしても反射性、光輝性等の増大に影響が少ない上に、蒸着をもって作成する経済的得策も少なくなる。これによって、インク、塗料等に使用したときに金属光沢性、光輝性に優れたまたは機能性を付与し得るインク、塗料等の

被覆材または金属光沢性、光輝性に優れたまたは機能性を付与し得る充填材となる。本発明における厚さが０．００５μｍ～３μｍの無機蒸着薄膜層（Ｃ）のうち無機化合物蒸着薄膜層としては、アルミニウム、錫、インジウム、チタン、ケイ素、亜鉛の酸化物、炭化物、窒化物、硫化物の単体化合物または複合化合物の少なくとも一種が適宜選択使用される。また、前記例示のものの中から一種または二種以上を適宜選定し一層または二層以上にして使用してもよい。これらの無機化合物蒸着薄膜層の厚さは、０．００５μｍ～３μｍの範囲が好ましく、０．００５μｍに満たないときは、機能性において（例えば導電性、抗菌性等の機能性）その性能は乏しく、３μｍを超えるときは、これ以上厚さを大きくしても機能性の増大に影響が少ない上に、蒸着をもって作成する経済的得策も少なくなる。本発明によって得られた無機箔粉を使用することで、インク、塗料等または充填材に使用したときに機能性に優れたインク、塗料等の被覆材、または充填材となる。本発明においてより好ましい実施形態としては、経済性などの観点から、無機蒸着薄膜層として金属アルミニウムを選択使用する製造方法とそれによって製造されたアルミニウム主体の箔粉が挙げられる。

【０００９】本発明は、基材としてのポリオレフィンフイルム（Ａ）の面上に、厚さが０．００５μｍ～３μｍの無機蒸着薄膜層（Ｃ）を設け、該無機蒸着薄膜層（Ｃ）を剥離し、剥離された層（Ｃ）を破砕する無機箔粉の製法で、該ポリオレフィンフイルム上に添加物（Ｂ）の層を形成し、該層を剥離層として利用することを特徴とする無機箔粉の製造方法であり、前記無機蒸着薄膜層（Ｃ）を剥離する方法としては、形成された添加物（Ｂ）の層を該化合物が溶解およびまたは分散する溶媒等で処理し剥離する方法と機械的に剥離する方法、基材を超音波や低周波等の振動付与、延伸、収縮等の機械的処理等をして剥離する方法等が適宜選択併用使用される。これらの剥離方法において、ポリオレフィンフイルム（Ａ）と無機蒸着薄膜層（Ｃ）との積層体を、折り曲げる、叩く、かきとる、擦過する、急冷急加熱等の熱ショックを与える等の方法で、該無機蒸着薄膜層（Ｃ）にクラックを生じせしめる等の補助工程を付加せしめてもよい。得られた無機蒸着薄膜層（Ｃ）を粉砕する方法としては、特に限定されないが、超音波や可聴音波や高周波、低周波等の振動付与、ジェットミル、ボールミル、リングロールミル、ハンマーミル、チューブミル等が挙げられる。この粉砕を水中または溶媒中で行ってもよくその際、アルコール等の粘度調節材、乾燥促進材、沈降安定剤、界面活性剤等を同時に使用して粉砕してもよい。さらに特に粉砕を積極的に実施しなくても、例えば超音波処理を剥離時に併用する時等には剥離性フイルムから剥離することだけで、積層体が微細な非定型偏平片およびまたは半定形偏平片となる場合もある。

【００１０】本発明においては、必要に応じて無機蒸着薄膜層（Ｃ）の上層または下層に、厚さが０．００５μｍ～１．０μｍの透明無機薄膜塗布層を設けてもよく、透明無機薄膜塗布層の材料としては、ケイ素酸化物、アルミニウム酸化物、インジウム酸化物、錫酸化物等や、ケイ素－酸素結合含有無機物を形成するケイ素アルコキシド、チタンアルコキシド、ジルコニウムアルコキシド、等の金属アルコキシド、水ガラス、ポリシラザン、ポリフォスファゼン等を塗布し、触媒作用、加熱、紫外線照射などによって透明無機薄膜塗布層を形成するものであれば特に限定されないが、透明無機薄膜塗布層としての透明性、無機蒸着薄膜層（Ｃ）の保護性から、ケイ素－酸素結合含有無機物を形成するもの、アルミニウム－酸素結合含有無機物を形成するもの、ジルコニウム－酸素結合含有無機物を形成するもの、チタン－酸素結合含有無機物を形成するものが好ましい。これらの透明無機薄膜塗布層を形成するアルコキシドなどを水やアルコールに溶解または分散して塗布し、触媒作用、加熱、紫外線照射などによって透明無機薄膜塗布層を形成する際、金属酸化物の微細粉体、例えばシリカ、酸化チタン、酸化アルミニウム、酸化ジルコニウム、酸化錫、酸化インジウム、酸化マグネシウムを添加含有せしめてもよく、これらの金属－酸素結合を形成するアルコキシド等と金属酸化物の微細粉体との金属が同一でもよく異種でもよく、２種以上の金属から選ばれたものであってもよい。さらに、透明無機薄膜塗布層を形成するアルコキシドなどを水やアルコールに溶解または分散して塗布し、触媒作用、加熱、紫外線照射などによって透明無機薄膜塗布層を形成する際に、本発明の目的、剥離性を損なわない限りにおいて、有機ポリマーなどのバインダー、色素や染料、レベリング剤、等を含有せしめてもよく、かかる色素や染料などを該透明無機薄膜塗布層に含有せしめることは蒸着などの乾式製膜法においては困難であり、塗布によって初めて達成でき、金属光輝性への耐熱色相の付与等が可能となる。

【００１１】

【実施例】参考例１

厚さ２０ミクロンの延伸ポリプロピレンフイルム（東洋紡績株式会社製のパイレンフイルム；Ｐ３１６２＃２０と同Ｐ２２４１＃２０の２種）を製膜後２週間放置したものおよび製膜後１週間放置したものを６０℃で２４時間エージングしたものを用意した。（なお、Ｐ３１６２＃２０の場合はヒートシール面以外の面を使用した。）

＊＊実施例１

参考例１記載における厚さ２０ミクロンの延伸ポリプロピレンフイルム４種の表面に（使用面も参考例１記載と同じ）、金属アルミニウムを蒸着し厚さ２５０Å、５００Å、７５０Åの各厚さで金属アルミニウム蒸着薄膜層を形成した蒸着体を得、この蒸着体を６０℃で２４時間エージングした。エージング後の蒸着体を１０ｃｍ×１

０ｃｍの大きさに裁断し、超音波洗浄機に大量の酢酸エチルと共に入れ、超音波をかけると各厚さの蒸着体、４種フイルム共に、数秒で殆ど全ての金属アルミニウム蒸着薄膜層が剥離した。さらに超音波処理を続行したところ、各厚さの一辺の最大大きさが平均で５～５０μｍ程度の特殊光輝性の美麗な金属箔粉を得ることができた。

【００１２】＊＊実施例２

参考例１記載における厚さ２０ミクロンの延伸ポリプロピレンフイルム４種の表面に（使用面も参考例１記載と同じ）、金属アルミニウムを蒸着し厚さ２５０Å、５００Å、７５０Åの各厚さで金属アルミニウム蒸着薄膜層を形成した蒸着体を得、この蒸着体を６０℃で２４時間エージングした。エージング後の蒸着体を５％延伸し金属アルミニウム蒸着薄膜層にクラックを生じさせた後、１０ｃｍ×１０ｃｍの大きさに裁断し、ケロシンを満たした容器に入れ、ゆっくりと撹拌すると、各厚さの蒸着体、４種フイルム共に、数秒で全ての金属アルミニウム蒸着薄膜層金属が剥離した。さらに該容器内で超音波処理を実行したところ、各厚さの一辺の最大大きさが平均で５～５０μｍ程度の特殊光輝性の美麗な金属箔粉を得ることができた。

【００１３】比較例１

参考例１での厚さ２０ミクロンの延伸ポリプロピレンフイルム（東洋紡績株式会社製のパイレンフイルム；Ｐ３１６２＃２０と同Ｐ２２４１＃２０の２種）を製膜後２週間放置したものを酢酸エチルで洗浄処理して表層に部分的に形成された添加物層を除去し、該洗浄処理したフイルム面に、金属アルミニウムを蒸着して厚さ２５０Å、５００Å、７５０Åの各厚さで金属アルミニウム蒸着薄膜層を形成した蒸着体を得、この得られた蒸着体を１０ｃｍ×１０ｃｍの大きさに裁断し、超音波洗浄機に大量の酢酸エチルと共に入れ、超音波をかけたが各厚さの蒸着体、２種フイルム共に、２分後においても、金属アルミニウム蒸着薄膜層が殆ど剥離しなかった。

【００１４】比較例２

参考例１で得た２種の６０℃で２４時間エージング処理済みの厚さ２０ミクロンの延伸ポリプロピレンフイルムを酢酸エチルで洗浄処理して表層に形成された添加物層を除去し、該洗浄処理したフイルム面に、金属アルミニウムを蒸着して厚さ２５０Å、５００Å、７５０Åの各厚さで金属アルミニウム蒸着薄膜層を形成した蒸着体を得、この得られた蒸着体を１０ｃｍ×１０ｃｍの大きさに裁断し、超音波洗浄機に大量の酢酸エチルと共に入れ、超音波をかけたが各厚さの蒸着体の２種フイルム共に、２分後においても、金属アルミニウム蒸着薄膜層が殆ど剥離しなかった。

【００１５】＊＊実施例３

比較例１記載における酢酸エチルで洗浄処理して表層に形成された添加物層を除去した延伸ポリプロピレンフイルム２種の表面に（使用面も参考例１記載と同じ）、金属アルミニウムを蒸着して厚さ２５０Å、５００Å、７５０Åの各厚さで金属アルミニウム蒸着薄膜層を形成した蒸着体を得、この蒸着体を６０℃で３６時間エージングした。エージング後の蒸着体を５％延伸し金属アルミニウム蒸着薄膜層にクラックを生じさせた後、１０ｃｍ×１０ｃｍの大きさに裁断し、ケロシンを満たした容器に入れ、ゆっくりと撹拌すると、各厚さの蒸着体、２種フイルム共に、数秒で大半の（面積で約８５％の）金属アルミニウム蒸着薄膜層が剥離した。さらに該容器内で超音波処理を実行したところ、各厚さの一辺の最大大きさが平均で５～５０μｍ程度の特殊光輝性の美麗な金属箔粉を得ることができた。

【００１６】

【発明の効果】本発明によって、極めて薄い無機蒸着薄膜層からなる箔粉を得るに際し、無機蒸着薄膜層が基材であるポリオレフィンフイルムから容易に剥離することができ且つ異物としての混入物の少ないしかも異物の除去の簡単な金属箔粉を経済的に得ることができ、得られた無機箔粉を使用することで、インク、塗料等の被覆材に金属光輝性または各種の機能を付与し得、紙や布帛やフイルム等への印刷、塗布、自動車の外装、壁材等の建具の外装、化粧品容器等の容器外装等に使用でき、家具、化粧品、筆記具、日用品、通信機器、電気製品、繊維製品、装身具等の外装および充填による機能付与に使用し得ることができた。

フロントページの続き

Ｆターム(参考)　4J037 AA04 EE29 FF09
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BA05 BA07 BA10 BA12 BA15
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METHOD FOR MANUFACTURING INORGANIC FOIL POWDER AND INORGANIC FOIL POWDER

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Inventor(s): NAGAI SHINGO; ARAKI SHUICHI; MURAKAMI YOSHINAGA
Applicant(s): OIKE KOGYO KK
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Abstract of **JP 2002004031 (A)**

PROBLEM TO BE SOLVED: To provide extremely thin inorganic foil powder such as metallic foil powder having excellent metallic luster or the like and to provide its production method. SOLUTION: In this method, a layer of added substance such as a lubricant is widely deposited on the face of a polyolefin film, an inorganic vapor deposited thin film is deposited and laminated on the layer of the added substance, and thereafter, by utilizing the lubricity, solubility or peelability of the added substance, solvent treatment with a solvent or the like or mechanical peeling is performed to easily peel and pulverize the inorganic vapor deposited thin film layer, and the extremely thin inorganic foil powder such as metallic foil powder having excellent metallic luster or the like is obtained thereby.

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(54) METHOD FOR MANUFACTURING INORGANIC FOIL POWDER AND INORGANIC FOIL POWDER

(57)Abstract:
PROBLEM TO BE SOLVED: To provide extremely thin inorganic foil powder such as metallic foil powder having excellent metallic luster or the like and to provide its production method.
SOLUTION: In this method, a layer of added substance such as a lubricant is widely deposited on the face of a polyolefin film, an inorganic vapor deposited thin film is deposited and laminated on the layer of the added substance, and thereafter, by utilizing the lubricity, solubility or peelability of the added substance, solvent treatment with a solvent or the like or mechanical peeling is performed to easily peel and pulverize the inorganic vapor deposited thin film layer, and the extremely thin inorganic foil powder such as metallic foil powder having excellent metallic luster or the like is obtained thereby.

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Applicant: Oike Kogyo KK

Title of Invention

Method for Manufacturing Inorganic Foil Powder and Inorganic Foil Powder

Detailed Explanation of Invention

——※——※——※——※——※——※——※——※——

[0005]

[Means for solving the problem]

The invention provides an inorganic foil powder and its producing method. Said producing method has processes that

- an inorganic vapor deposited thin film layer with a thickness of 0.005μm-3μm (C) is deposited on the face of a polyolefin film layer (A),
- the inorganic vapor deposited thin film layer (C) is peeled,
- the peeled layer (C) is pulverized,

which is characterized by

- a layer of added substance (B) which is included previously in the polyolefin film layer (A) and/or the layer of added substance (B) are/is formed as a peeled layer between the face of the polyolefin film layer (A) and the inorganic vapor deposited thin film layer (C),
- the inorganic vapor deposited thin film layer (C) is made of

at least one sort of simple metallic substance or their alloy and mixture of Al, Ag, Au, Ni, Cr, Sn, Cu, Zn, In, Ti or Mg,

- the inorganic vapor deposited thin film layer (C) is made of at least one sort of one or more than one compound of oxide, carbide, nitride, sulphide, or halide of Al, Sn, In, Ti, Si, Zn, or Mg.

Said inorganic foil powder is characterized by having a thickness of 0.005μm-3μm and an average size of 2μm-150μm, produced by one of said producing method.

[0011]

**Experimental example 1

Metallic aluminium was vapor deposited on the face of four kinds of oriented polypropylene film with a thickness of 20μm. Vapor deposited body is obtained, which forms metallic aluminium vapor deposited thin film with three thickness types of 250Å, 500Å and 750Å. This vapor deposited body was aged at 60 degrees for 24 hours and was cut into a size of 10cm x 10cm. They were put with a large amount of ethyl acetate into an ultrasonic cleaner. When ultrasonic was applied, almost the entire metallic aluminium vapor deposited thin film layer was peeled in a few seconds, every thickness of vapor deposited bodies and all of the four kinds of film had the same reaction. With further application of ultrasonic treatment, excellent metallic foil powder having a peculiar luster with maximum side 5-50μm long on average could be obtained.

* NOTICES *

DETAILED DESCRIPTION

[Detailed Description of the Invention]
[0001]
[Field of the Invention]This invention relates to the manufacturing method of the inorganic foil powder which is a crushing atypical flat piece of an inorganic deposition thin film layer, or a crushing half fixed form (for example, rectangle) flat piece at least.
By using this inorganic foil powder as covering material and bulking agents, such as ink and a paint, printing and drawing which were expressed by the subject -- or the surface whole -- or it is related with the process of inorganic foil powder and inorganic foil powder which can give the function in which this foil powder holds [in the case of metallic foil powder] metallic luster etc. at a filler in the case of metallic-compounds foil powder.

[0002]
[Description of the Prior Art]as metallic foil powder -- ** -- the spall of fines, i.e., the spreading foil obtained by spreading, such as the precious metals known for many years,. ** The metallic foil powder which is a condensation detailed piece at the time of vacuum evaporation in recent years, and a spall of ** metallic foil, ** The metal powder etc. which are the crushing wafers of the metal deposition foil by which one side or both sides of the statement were covered by resin from vacuum evaporation metal to the decision glitter and ** JP,53-35135,B on a film given in JP,5-77643,B are known.
[0003]
[Problem(s) to be Solved by the Invention]the metallic foil or metal powder of said former -- ** -- the spall of fines, i.e., the spreading foil obtained by spreading, such as the precious metals known for many years, although used abundantly from the corrosion resistance of the precious metals, beautiful, and a detailed thing, The specific gravity which is a high cost and cannot be produced in large quantities from the process is too large, and it has a technical problem of being easy to precipitate in a carrier fluid body.
** Although the condensation detailed piece at the time of vacuum evaporation in recent years is attractive in the detail, it has technical problems, such as the high cost and it being too detailed, and having a stake to obtain beautiful [of metal photoluminescence] and having a difficulty also in handling.
[0004]** Although the metallic foil powder which is a spall of a metallic foil can be comparatively obtained by low cost, has the performance of a grade in which it has also set to photoluminescence and is used abundantly, That in which the size is tens of micrometers - hundreds of micrometers is most, and when smoothing of a spreading side and high photoluminescence are required more, it has a technical problem of being unable to respond.
** Although the decision glitter from the vacuum evaporation metal on the film of a statement is used for JP,5-77643,B from the low cost in a specific use, a size is about several millimeters in size, and a use is limited.
** the metal powder which is a crushing wafer of the metal deposition foil with which one side or both sides of the statement were covered with resin by JP,53-35135,B -- the size, although comparatively used abundantly by the high photoluminescence from low cost, Since it is covered with resin, it becomes thick for resin layer thickness, and there is a limit in ** and heat resistance in the point of thickness. Although the method of exfoliating, crushing a metallic vapor deposition thin film layer from a film by forming the layer of good solubility polymer on a film furthermore, forming a metallic vapor deposition thin film layer, and carrying out dissolution treatment of the layer of good solubility polymer, and obtaining metallic foil powder is also known, Technical problems, such as taking great cost and/or the big-ticket equipment for dangerous thief treatment to remove the layer of good solubility polymer adhering to this foil powder from metallic foil powder, were held. the technical problem in which said conventional metallic-foil-powder process holds this invention -- it is going to

solve -- the foil powder which it was a thing and thickness was 3 micrometers or less, and excelled [size] in the photoluminescence of several micrometers - about 100 micrometers, and excelled [size] in heat resistance -- it is going to provide -- it is a thing.

[0005]

[Means for Solving the Problem]Namely, in a manufacturing method of inorganic foil powder which this invention provides an inorganic deposition thin film layer (C) whose thickness is 0.005 micrometer - 3 micrometers on a field of a polyolefin film (A), exfoliates this inorganic deposition thin film layer (C), and crushes a layer (C) which exfoliated, A layer of an additive (B) which carries out content maintenance beforehand to this polyolefin film (A), And/or, it is a manufacturing method of inorganic foil powder making a layer of this same additive (B) form as stratum disjunctum between a field of a polyolefin film (A), and an inorganic deposition thin film layer (C), An inorganic deposition thin film layer (C) Aluminum, silver, gold, nickel, chromium, It is a manufacturing method of the aforementioned inorganic foil powder of tin, copper, zinc, indium, titanium, simple substance metal of magnesium or these alloys, and those mixtures which consists of kinds at least, An inorganic deposition thin film layer (C) Aluminum, tin, indium, titanium, Thickness which is a manufacturing method of the aforementioned inorganic foil powder of silicon, zinc, an oxide of magnesium, carbide, a nitride, a sulfide, a simple substance compound of a halogenide, or a conjugated compound which consists of kinds at least, and was manufactured by said which manufacturing method further again 0.005 micrometer - 3 micrometers, It is inorganic foil powder, wherein sizes are an average of 2 micrometers - 150 micrometers.

[0006]

[Embodiment of the Invention]As a polyolefin film (A) in this invention, Especially if it is a polyolefin film of independence nature which can bear heating of a deposition process, a vacuum, stress, etc., will not be limited, but although gay polyolefin films, the films from these polyolefin system copolymers, etc., such as polyethylene and polypropylene, are mentioned, A polypropylene system film and a polyethylene system film can use it preferably especially. The thickness of these films will be 4 micrometers to 100 micrometers, what is less than 4 micrometers is dealt with at a process etc., a sex has many problems, if it exceeds 100 micrometers, it will become lacking in pliability and its a problem will increase in exfoliation etc. As these polyolefin films (A), the film of a monolayer may be sufficient, for example, it may be a double layer film by a coextrusion process. These films do not receive the limitation in particular by processes, such as no extending, uniaxial stretching, biaxial stretching, and weak uniaxial stretching.

[0007]As a polyolefin film (A) in this invention, The additive (B) as lubricant, sticking inhibitor, an antifogger, a spray for preventing static electricity, etc. is made to contain and hold by addition, content and spraying, or other means by this film, and this film has this additive in 0.01 to 2.0% of the weight of the range. As a layer of an additive (B), on a polyolefin film (A), bleed out, It is what transpires at the time of formation of the inorganic deposition thin film layer (C) whose thickness which is formed by spreading etc. and formed on the layer is 0.005 micrometer - 3 micrometers, or carries out thermal denaturation, and does not interfere with manufacture of metallic foil powder, a molecular weight -- 5000 or less thing -- a 1000 or less-about compound being preferred, and still more preferably, 3000 or less preferably, What is necessary is just what exfoliation by the dissolution from the polyolefin film (A) of an inorganic deposition thin film layer (C), etc. tends to carry out, Although not limited in particular, what uses these denaturation things, such as fatty acid, metal fatty acid, a low molecule surface-active agent, polyoxy alkylene glycol, paraffin, an acid amide, and fatty acid ester, as the main ingredients is mentioned, and the denaturation thing of these and an inorganic compound, a mixture, etc. can be used. It can set to this invention "the layer of the additive (B) which carries out content maintenance beforehand to this polyolefin film (A), And/or, the thing made to form the layer of this same additive (B) as stratum disjunctum between the field of a polyolefin film (A), and an inorganic deposition thin film layer (C)", Although not limited in particular, before vapor-depositing on a film, it may form by spreading, spraying, etc., The polymer of the same kind which made the additive (B) contain may be used as the film laminated on the polymer of the same kind which hardly contains this additive (B) by co-extrusion etc. (double layer), and what is called a thing that carries out bleed out may be used, The method of scouring to the base material film which is a polyolefin film (A) beforehand, and this film surface carrying out bleed out before vacuum evaporation or after vacuum evaporation by processing with the passage of time, heat temporality processing, heat treatment, etc., etc. are mentioned. Scour to the base material film which is a polyolefin film (A) beforehand, and before vacuum evaporation or after vacuum evaporation, In what is called the method of carrying out bleed out to this film surface by processing with the passage of time, heat temporality processing, heat treatment, etc., After vapor-depositing an inorganic deposition thin film layer, what is called the method of carrying out bleed out is adopted

more preferably, also in it, the heat temporality processing accompanied by heating at not less than 40 ** will be preferred, and this additive layer will be uniformly formed in this film surface. In [perform processing with the passage of time, heat temporality processing, and heat treatment before vacuum evaporation, and] what is called the method of carrying out bleed out a content additive to this film surface, So that it may be preferred that this additive layer is uniformly formed in this film surface and it may become it is desirable and easy [exfoliation of an inorganic deposition thin film layer (C)] not less than 50% of area [it], Decline in being formed to not less than 50% and yield [in / what is not less than 85% still more preferably not less than 70% more preferably, and exfoliates easily in 50% or less of case serves as 50% or less of surface integral, and / foil powder manufacture], and the rise of cost will be caused. Therefore, it includes that this invention carries out selection use of the polyolefin film (A) in which the layer of a minute partial additive (B) is not formed by the temporality by the neglect after manufacture, and the layer of the additive (B) was formed in the above mentioned large area.

[0008]The formation method of the inorganic deposition thin film layer (C) whose thickness in this invention is 0.005 micrometer - 3 micrometers will not be limited especially if based on vacuum evaporation, but it should just carry out selection use suitably from heating vacuum evaporation, sputtering, etc. The monolayer of a metallic vapor deposition thin film layer may be sufficient as these inorganic deposition thin film layers (C), the double layer of different species or the same kind may be sufficient as them, the monolayer of an inorganic compound deposition thin film may be sufficient as them, and the double layer of different species or the same kind may be sufficient as them. Lamination with a metallic vapor deposition thin film layer and an inorganic compound deposition thin film layer may be sufficient. The metal used as a metallic vapor deposition thin film layer among inorganic deposition thin film layers (C), Especially if it has functions, such as having metallic luster, it is not limited, but even if there are few simple substance metal of aluminum, silver, gold, nickel, chromium, tin, zinc, indium, and titanium ** or these alloys, and those mixtures, selection use of more than a kind is carried out suitably. moreover -- selecting a kind or two sorts or more suitably from the things of said illustration -- much more -- or it may be used, carrying out more than a bilayer. When the thickness of these metallic vapor deposition thin film layers has the preferred range of 0.005 micrometer - 3 micrometers and it is less than 0.005 micrometer, When the performance is scarce and it exceeds [in / again / on photoluminescence and / functionality] 3 micrometers, even if it enlarges thickness more, in increase of reflexibility, photoluminescence, etc., there is little influence and the economical best policy which has and creates vacuum evaporation also decreases. It becomes a filler which can be excellent or give functionality to the covering material, such as ink and a paint, or metallic luster nature, and photoluminescence which metallic luster nature and photoluminescence were excellent, or can give functionality to by this when it is used for ink, a paint, etc. Among the inorganic deposition thin film layers (C) whose thickness in this invention is 0.005 micrometer - 3 micrometers, as an inorganic compound deposition thin film layer, even if there are few aluminum, tin, indium, titanium, silicon, a zincky oxide, carbide, nitrides, simple substance compounds of a sulfide, or conjugated compounds, selection use of a kind is carried out suitably. moreover -- selecting a kind or two sorts or more suitably from the things of said illustration -- much more -- or it may be used, carrying out more than a bilayer. When the thickness of these inorganic compound deposition thin film layers has the preferred range of 0.005 micrometer - 3 micrometers and it is less than 0.005 micrometer, When the (for example, functionality, such as conductivity and antibacterial properties) performance is scarce and it exceeds 3 micrometers in functionality, even if it enlarges thickness more, in increase of functionality, there is little influence and the economical best policy which has and creates vacuum evaporation also decreases. By using the inorganic foil powder obtained by this invention, when it is used for fillers, such as ink and a paint, it becomes covering material, such as ink excellent in functionality, and a paint, or a filler. In this invention, the foil powder of the manufacturing method which carries out selection use of the metallic aluminum as an inorganic deposition thin film layer, and the aluminum subject manufactured by it is mentioned from viewpoints of economical efficiency etc. as a more desirable embodiment.

[0009]This invention is a process of the inorganic foil powder which provides the inorganic deposition thin film layer (C) whose thickness is 0.005 micrometer - 3 micrometers on the field of the polyolefin film (A) as a substrate, exfoliates this inorganic deposition thin film layer (C), and crushes the layer (C) which exfoliated, It is a manufacturing method of the inorganic foil powder forming the layer of an additive (B) on this polyolefin film, and using this layer as stratum disjunctum, As a method of exfoliating, said inorganic deposition thin film layer (C), Selection concomitant use use of the method of processing the layer of the formed additive (B) with the solvent etc. which this compound dissolves and/or distributes, and exfoliating, the method of exfoliating

mechanically, the method of carrying out mechanical processes, such as oscillating grant of an ultrasonic wave, a low frequency wave, etc., extension, and contraction, etc., and exfoliating a substrate, etc. is carried out suitably. By giving heat shocks [which scrape] which bend the layered product of a polyolefin film (A) and an inorganic deposition thin film layer (C), to strike, to write, such as quenching sudden heating, in these peeling methods etc., Auxiliary processes, such as producing and cheating out of a crack to this inorganic deposition thin film layer (C), may be made to add. Especially as a method of grinding the obtained inorganic deposition thin film layer (C), although not limited, oscillating grant of an ultrasonic wave, an audible sound wave, high frequency, a low frequency wave, etc., etc., a jet mill, a ball mill, a ring roll mill, a hammermill, a tube mill, etc. are mentioned. This grinding may be performed underwater or in a solvent, and it may grind in that case, using simultaneously viscosity regulation material, such as alcohol, dry promotion material, sedimentation stabilizer, a surface-active agent, etc. Even if it does not grind positively further in particular, a layered product may serve as a detailed atypical flat piece and/or a half-fixed form flat piece only by exfoliating from a detachability film, when using ultrasonication together at the time of exfoliation, for example.

[0010]In this invention, if needed in the upper layer or the lower layer of an inorganic deposition thin film layer (C). May provide the transparent inorganic thin film coating layer whose thickness is 0.005 micrometer - 1.0 micrometer, and as a material of a transparent inorganic thin film coating layer, A silicon oxide, an aluminum oxide, an indium oxidation thing, a stannic acid ghost, etc., The silicon alkoxide, the titanium alkoxide which form a silicon-oxygen bond content inorganic substance, Metal alkoxides, such as zirconium alkoxide, water glass, polysilazane, Especially if polyphosphazene etc. are applied and a transparent inorganic thin film coating layer is formed by a catalysis, heating, UV irradiation, etc., will not be limited, but. From the transparency as a transparent inorganic thin film coating layer, and the protection nature of an inorganic deposition thin film layer (C). What forms a silicon-oxygen bond content inorganic substance, the thing which forms an aluminum oxygen bond content inorganic substance, the thing which forms a zirconic acid matter joint content inorganic substance, and the thing which forms a titanic acid matter joint content inorganic substance are preferred. Dissolve or distribute and the alkoxide etc. which form these transparent inorganic thin film coating layers are applied to water or alcohol, When a transparent inorganic thin film coating layer is formed by a catalysis, heating, UV irradiation, etc., The detailed granular material of a metallic oxide, for example, silica, titanium oxide, an aluminum oxide, Addition content of zirconium oxide, tin oxide, indium oxide, and the magnesium oxide is carried out, the metal of the alkoxide etc. which form these metal-oxygen bonds, and the detailed granular material of a metallic oxide may be the same, and different species may be sufficient as it, and it may be chosen from two or more sorts of metal. Dissolve or distribute and the alkoxide etc. which form a transparent inorganic thin film coating layer are applied to water or alcohol, When forming a transparent inorganic thin film coating layer by a catalysis, heating, UV irradiation, etc., unless the purpose of this invention and detachability are spoiled, Binders, such as organic polymer, coloring matter, a color, a leveling agent, etc. may be made to contain, It is difficult in the dry type producing-film methods, such as vacuum evaporation, to make this transparent inorganic thin film coating layer contain this coloring matter, color, etc., it will not be able to attain without spreading, and grant of the heat-resistant hue to metal photoluminescence, etc. are attained.

[0011]

[Example]What aged at 60 ** what was neglected for one week after the thing neglected for two weeks after producing a stretched polypropylene film (two sorts, pi-wren film-3162#20 and the P2241#20 by Toyobo Co., Ltd.) with a reference example 1 thickness of 20 microns and film production for 24 hours was prepared. (In addition, in the case of P3162#20, fields other than a heat-sealing side were used.)

On the surface of four sorts of stretched polypropylene films with a thickness [in * example 1 reference-example 1 statement] of 20 microns, * (A use side is the same as reference example 1 statement), The vacuum evaporation object which vapor-deposited metallic aluminum and formed the metallic aluminum deposition thin film layer by each thickness of 250 A in thickness, 500 A, and 750 A was acquired, and this vacuum evaporation object was aged at 60 ** for 24 hours. The vacuum evaporation object after aging was cut out in size of 10 cm x 10 cm, and it put into the ultrasonic washing machine with a lot of ethyl acetate, and when the ultrasonic wave was applied, in the vacuum evaporation object of each thickness, and a four-sort film, almost all metallic aluminum deposition thin film layers exfoliated in several seconds. When ultrasonication was furthermore continued, the maximum size of one side of each thickness was able to obtain the about 5--50--micrometer beautiful metallic foil powder of special photoluminescence on the average.

[0012]On the surface of four sorts of stretched polypropylene films with a thickness [in * example 2 reference-example 1 statement] of 20 microns, * (A use side is the same as reference example 1 statement), The vacuum

evaporation object which vapor-deposited metallic aluminum and formed the metallic aluminum deposition thin film layer by each thickness of 250 A in thickness, 500 A, and 750 A was acquired, and this vacuum evaporation object was aged at 60 ** for 24 hours. If it judges in size of 10 cm x 10 cm, it puts into the container which filled kerosene and it stirs slowly after extending the vacuum evaporation object after aging 5% and making a metallic aluminum deposition thin film layer produce a crack, In the vacuum evaporation object of each thickness, and a four-sort film, all the metallic aluminum deposition thin film layer metal exfoliated in several seconds. When ultrasonication was furthermore performed within this container, the maximum size of one side of each thickness was able to obtain the about 5-50-micrometer beautiful metallic foil powder of special photoluminescence on the average.

[0013]The additive layer which carried out washing processing of what was neglected for two weeks after producing a stretched polypropylene film (two sorts, pi-wren film-3162#20 and the P2241#20 by Toyobo Co., Ltd.) with a thickness [in the comparative example 1 reference example 1] of 20 microns with ethyl acetate, and was selectively formed in the surface is removed, To this film plane that carried out washing processing, vapor-deposit metallic aluminum, and to it 250 A in thickness. The vacuum evaporation object which formed the metallic aluminum deposition thin film layer by each thickness of 500 A and 750 A is acquired, This acquired vacuum evaporation object was cut out in size of 10 cm x 10 cm, and it put into the ultrasonic washing machine with a lot of ethyl acetate, and although the ultrasonic wave was applied, 2 minutes afterward [film / the vacuum evaporation object of each thickness, and / two sort], a metallic aluminum deposition thin film layer hardly exfoliated.

[0014]The additive layer which carried out washing processing of the stretched polypropylene film with a thickness [finishing / aging processing] of 20 microns with ethyl acetate for 24 hours at two sorts of 60 ** obtained by the comparative example 2 reference example 1, and was formed in the surface is removed, To this film plane that carried out washing processing, vapor-deposit metallic aluminum, and to it 250 A in thickness. The vacuum evaporation object which formed the metallic aluminum deposition thin film layer by each thickness of 500 A and 750 A is acquired, This acquired vacuum evaporation object was cut out in size of 10 cm x 10 cm, and it put into the ultrasonic washing machine with a lot of ethyl acetate, and although the ultrasonic wave was applied, 2 minutes afterward [film / of the vacuum evaporation object of each thickness / two sort], a metallic aluminum deposition thin film layer hardly exfoliated.

[0015]The additive layer which carried out washing processing with the ethyl acetate in * example 3 comparative-example 1 statement, and was formed in the surface on the surface of two sorts of removed stretched polypropylene films * (A use side is the same as reference example 1 statement), The vacuum evaporation object which vapor-deposited metallic aluminum and formed the metallic aluminum deposition thin film layer by each thickness of 250 A in thickness, 500 A, and 750 A was acquired, and this vacuum evaporation object was aged at 60 ** for 36 hours. If it judges in size of 10 cm x 10 cm, it puts into the container which filled kerosene and it stirs slowly after extending the vacuum evaporation object after aging 5% and making a metallic aluminum deposition thin film layer produce a crack, In the vacuum evaporation object of each thickness, and a two-sort film, most metallic aluminum (it is about 85% in area) deposition thin film layers exfoliated in several seconds. When ultrasonication was furthermore performed within this container, the maximum size of one side of each thickness was able to obtain the about 5-50-micrometer beautiful metallic foil powder of special photoluminescence on the average.

[0016]

[Effect of the Invention]It faces obtaining the foil powder which consists of a very thin inorganic deposition thin film layer by this invention, an inorganic deposition thin film layer can exfoliate easily from the polyolefin film which is a substrate --- and the contaminant as a foreign matter -- it is few --- a deer can also obtain economically the easy metallic foil powder of removal of a foreign matter, and by using the obtained inorganic foil powder. The function of metal photoluminescence or various kinds can be given to covering material, such as ink and a paint, It can be used for the container exterior, such as printing to paper, a textile, a film, etc., spreading, the exterior of a car, the exterior of fittings, such as a wallplate, and a cosmetic container, etc., and was able to be used for the functional grant by the exterior, such as furniture, cosmetics, pens and pencils, daily needs, communication equipment, an electric product, textiles, and accessories, and restoration.

[Translation done.]